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DEFLAGRATION BEHAVIOR OF PBXN-109 AND COMPOSITION B AT HIGH PRESSURES AND TEMPERATURES

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ABSTRACT

We report deflagration rate measurements on PBXN-109 (RDX/Al/HTPB) and Composition B (RXD/TNT/wax) at pressures from 1,500 - 100,000 psi (10-700 MPa). This was done with the LLNL High Pressure Strand Burner, in which embedded wires are used to record the time-of-arrival of the burn front in the cylindrical sample as a function of pressure. The propellant samples are 6.4 mm in diameter and 6.4 mm long, with burn wires inserted between samples. Burning on the cylindrical surface is inhibited with an epoxy or polyurethane layer. With this direct measurement we do not have to account for product gas equation of state or heat losses in the system, and the burn wires allow detection of irregular burning. We report deflagration results for PBXN-109 as received, and also after it has been damaged by heating. The burn behavior of pristine PBXN-109 is very regular, and exhibits a reduction in pressure exponent from 1.32 to 0.85 at pressures above 20,000 psi (135 MPa). When PBXN-109 is thermally damaged by heating to 170-180°C, the deflagration rate is increased by more than a factor of 10. This appears to be a physical effect, as the faster burning may be explained by an increase in surface area. Our results with Composition B show an apparent 2nd order pressure dependence for initial deflagration, followed by deconsolidation and onset of very rapid and erratic burning. The deconsolidation may be the result of the TNT melting as heat flows into the sample.

INTRODUCTION

During the response of energetic materials to hazards such as thermal or mechanical stimuli, the initial low-level reaction releases sufficient energy to cause an increase in pressure and temperature, which then leads to acceleration of the reaction until a runaway condition is reached. Accurate knowledge of the reaction rates at conditions typical of those in accelerating reactions is necessary to understand and predict the violence of the ensuing explosion. Materials with reaction rates that are strongly accelerated by pressure and temperature generally give more violent responses than materials whose reaction rates are less sensitive to pressure and temperature. For explosives and propellants undergoing thermal explosion or mechanical impact, hydrodynamic calculations show pressures in the reacting material of several hundred MPa (several kbar) and higher. Therefore, the laminar burn rates of these propellants at these high pressures is needed for accurate prediction of reaction violence through computer simulation. ^{1, 2}

The reactions taking place during the final explosion are, in situations that do not involve shock stimuli, deflagrative in nature. Therefore, measurement of the deflagration rates at high pressures and temperatures provides the necessary information on the runaway reaction behavior. We generally measure deflagration behavior of pristine materials first, as this provides significant information on the material. However, inasmuch as the material undergoing runaway reaction has generally been damaged, either thermally or mechanically, the deflagration behavior of the damaged energetic material must also be characterized.

As part of DOE and DoD programs to develop a predictive capability for thermal explosion onset and violence, we measured the deflagration rate of PBXN-109 and of Composition B high pressures and temperatures. We report results with PBXN-109 from our strand burner at pressures of 1,500-100,000 psi

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(10-700 MPa), for both pristine and thermally-damaged samples. We also report results with Composition B over the pressure range of 800-30,000 psi (5-200 MPa). Finally, we relate our results to closed bomb data for PBXN-109 at pressures of 2,000-8,000 psi (14-55 MPa),³ and to results with Composition B using a strand burner at pressures of 300-1,500 psi (2-10 MPa)⁴ and using a closed bomb at pressures up to 45,000 psi (300 MPa).⁵

EXPERIMENTAL APPARATUS

HIGH PRESSURE STRAND BURNER

The LLNL high pressure strand burner, shown schematically in Figure 1, combines the features of a traditional closed-bomb burner with those of a traditional strand burner. The LLNL high-pressure strand burner contains a burning sample in a small volume, high-pressure chamber. We measure temporal pressure data and burn front time-of-arrival data to get the laminar burn rate for a range of pressures in one experiment. We use a pressure transducer and a load cell to measure the temporal pressure in the bomb, and detect the arrival of the burn front by the burning-through of thin wires embedded in the sample. High speed digital scopes capture the data for subsequent analysis. In contrast, with a standard closed-bomb burner, pressure in the combustion chamber is the only measurement; calculation of the burn rate requires accurate knowledge of the equation of state of the product gases and accurate treatment of heat losses. There is no measure of the surface regression rate to check combustion uniformity, so data from samples that burn erratically are particularly hard to interpret. The standard strand burner provides direct measurement of the surface regression rate in a large volume at constant pressure, giving only one pressure/rate data point in each experiment; furthermore, the large volume required for isobaric operation means that operation at high pressures is generally not practical.

The strand burner has a volume of about 75 cm³, and is designed to reach pressures of 1 GPa (150,000 psi). The pressure vessel body is built from two concentric shells with interference between them to put the inner shell in compression. The standard inner liner, and the top and bottom closures, are fabricated from hardened S-5 tool steel, which is very high strength but also very brittle and prone to corrosion. The S-5 tool steel was suitable for these experiments with PBXN-109 and Composition B explosives.

The burn sample, shown in Figure 2, is a cylinder 58 mm long and 6.4 mm in diameter, made of nine cylindrical propellant pellets stacked on end; silver burn wires (75 µm diameter) are inserted between each pair of pellets, in a groove in each pellet. After assembly, the cylindrical surface of the sample is coated with an inhibitor to prevent burning of this surface; this limits the burn front to the end of the cylinder, resulting in a laminar burn. The sample end is ignited by a thin HNS pressed pellet (30 mg), which is in turn ignited by a hot wire and ~110 mg of boron potassium nitrate.

For PBXN-109, the inhibitor was an epoxy (Epon 815 with RF 20 catalyst). With Composition B, this epoxy caused significant discoloration of the explosive surface, indicating some degree of incompatibility. Therefore, Halthane 88-2,a polyurethane adhesive compatible with Composition B, was used in all cases. For some tests a layer of the epoxy was coated onto the Halthane 88-2, but no significant differences were seen between runs with only the Halthane coating and the runs with the Halthane plus epoxy coating.

To conduct a measurement, the burn sample is inserted into the pressure vessel. The sample mounts into a pre-wired base that carries the signal wires through high-pressure feedthroughs in the bottom plug of the pressure vessel. The system is pressurized to the desired starting pressure (up to 400 MPa or 60,000 psi) with argon, and then burned while recording pressure and burn wire data. One noteworthy feature is the use of solid argon plugs as the remotely-actuated pressure isolation valves. Once the system is pressurized (using manual valves), it is isolated with solid argon plugs by immersing sections of the inlet and outlet tubing in liquid nitrogen. The manual valves are then opened prior to going to remote operation for the burn. Following the burn, the pressure is released by remotely removing the liquid nitrogen from the outlet tubing. The solid argon plugs provide a reliable remotely-actuated valve for high-pressure gas with no moving parts and no seals to maintain.

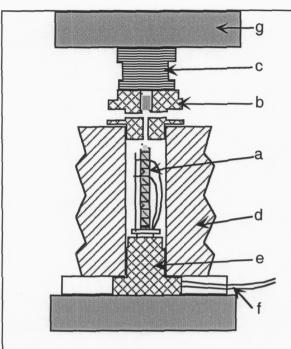


Figure 1. Schematic of strand burner. a - nine-segment burn sample, with burn wires between segments (only two wires shown for clarity) and ignitor on top; b - top plug with inlet and outlet ports and pressure transducer in center; c - load cell; d - pressure vessel; e - bottom plug with wire feedthroughs; f - signal wires to electronics; g - load frame (top and bottom)

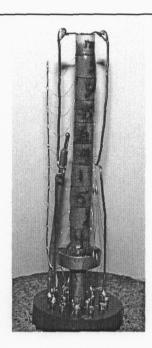


Figure 2. Sample holder and propellant sample. Center stack is sample, with burn wires between pellets. BKNO₃/HNS ignitor in cardboard tube is on top, with ignition wires leading to it. Thermocouple at left is used to monitor temperature inside pressure vessel. Individual pellets are 6.4 mm diameter and ~ 6.4 mm long

SAMPLE PREPARATION

PBXN-109 has a composition of 65% RDX, 21% aluminum, and 14% HTPB binder plasticized with DOA. The material used in this study was provided by Alice Atwood of NAWC China Lake from a master batch formulated at China Lake. We cut pellets of each material by hand, using a coring tool and razor knife, to get final pellet sizes of 6.4 mm diameter and ~ 6.4 mm length. A precision holder was used to maintain the ends of the pellets as flat and parallel as possible. Even with the tooling and careful handling, the samples were significantly less regular in shape than pressed or machined parts, with some irregularities in the diameter formed as the readily-deformable material was pressed through the coring tool. The bulk density of each pellet, as determined from mass and physical dimensions, ranged from 1.58-1.65 g/cc (95-99.3% of theoretical maximum density (TMD) of 1.662 g/cc). Given the uncertainties in physical dimensions, these results are consistent with the reported initial porosity of 0.5%.

We had two forms of Composition B (63% RDX, 36% TNT, 1% wax) samples. At LLNL, Composition B from batch C-377 was uniaxially pressed into pellets 6.4 mm in diameter and 6.4 mm in length. At the Army Research Laboratory, Composition B was cast and machined into samples of the same dimensions. All samples were uniform cylinders with smooth walls and parallel ends. The bulk density of pressed samples, as determined from mass and physical dimensions, ranged from 1.68-1.69 g/cc (97-98% of TMD of 1.732 g/cc). The bulk density of the cast samples ranged from 1.65-1.70 g/cc (95-98% TMD), with most in the range 1.67-1.69 g/cc (96-98% TMD).

RESULTS AND DISCUSSION

PBXN-109

Results from deflagration rate measurements with pristine PBXN-109 are shown in Figure 3. The deflagration behavior of PBXN-109 is remarkably stable over the entire pressure range, with data showing smooth and consistent increases with pressure within each run and from run to run. Also shown in Figure 3 are data at the lower end of our pressure range measured at NAWC. The LLNL and NAWC results show excellent agreement.

Our burn rate data in Figure 3 show a slope change around 20,000 psi (135 MPa). Power-law parameters and line fits are shown in Figure 3 for the low-pressure and high-pressure regions. The pressure exponent decreases significantly at pressures above 135 MPa from greater than 1 (1.32) to less than 1 (0.85).

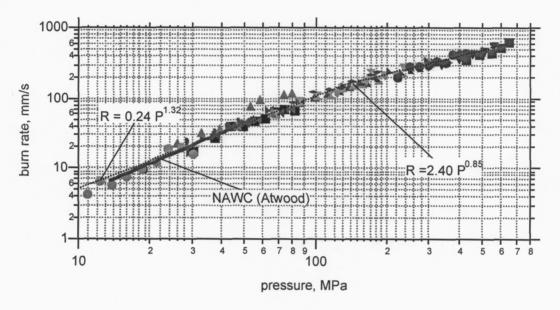


Figure 3. Deflagration rate data for pristine PBXN-109. Solid symbols are LLNL measurements. Each set of symbols represents data from one experiment. A fit to data from NAWC (Alice Atwood) is shown by the solid line. Power-law fits to low and high pressure regions are shown by the dotted lines.

The deflagration behavior of PBXN-109 after thermal damage is very important to the eventual violence of thermal explosion. This behavior is clearly dependent on the nature of the thermal damage. Instead of characterizing the extent and type of damage caused in PBXN-109 by high temperatures, we chose to thermally damage the PBXN-109 using time-temperature profiles close to those in actual NAWC-China Lake experiments. Following this approach, the deflagration behavior measured with these samples should directly reflect the expected deflagration behavior of the PBXN-109 during the thermal explosions in these tests. We note that this approach does not provide sufficient information to allow general prediction of the effect of thermal damage on deflagration; nonetheless, it does provide data sufficient to support the modeling of the specific NAWC tests.

The NAWC experiments were run with heating rates of 3°C/hr and 6°C/hr. We therefore heated samples at these heating rates to high temperatures (following an initial heating to 130°C followed by a thermal soak for ~ 30 minutes to allow thermal equilibration), and then ignited a deflagration if the sample had not already ignited. The deflagration rate data as measured by the burn wires are shown in Figure 4, below. We intended to heat the samples to a temperature below that of self-ignition and then deliberately ignite them; however, an error in the sample temperature calibration led to higher temperatures than

intended. The temperatures shown in Figure 4 represent the actual sample temperatures within a few degrees. Self-ignition or lack thereof is noted for each run in Figure 4 as well.

The data in Figure 4 show an increase in deflagration rates of up to 10-20 fold for thermally damaged samples. Interestingly, all samples showed this very fast deflagration for about the first $1/3^{rd}$ of the sample, with the remainder of the sample burning at rates about the same as or slightly slower than the pristine material. We do not have an explanation of this behavior, but it quite reproducible, as seen in Figure 4. This behavior is also shown in pressure-time data for these runs, with an example shown in Figure 5.

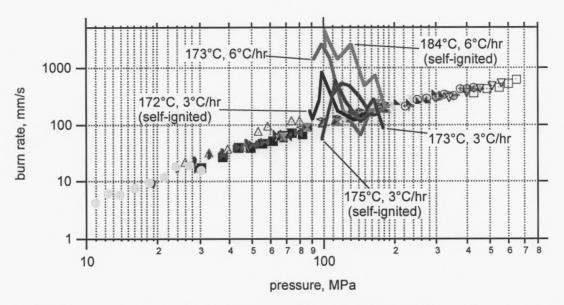


Figure 4. Deflagration rate data for thermally-damaged PBXN-109 (lines), compared with data for pristine samples (symbols). The thermal ramp rate and final sample temperature are shown for each run.

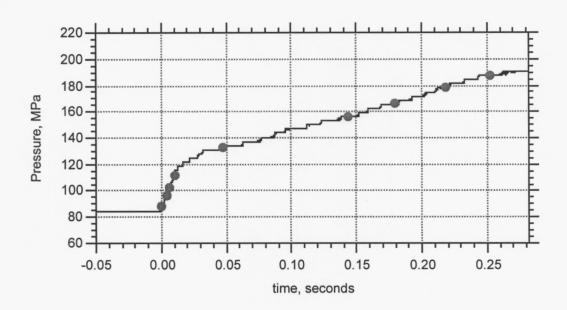


Figure 5. Pressure-time data for thermally-damaged PBXN-109, showing initial rapid deflagration followed by a significantly reduced burning rate. Markers show reporting times for embedded burn wires. Data are for sample heated to 173°C at 6°C/hr.

The concept of vivacity provides another way to quantify the increase in burn rate. Vivacity is defined as:

$$vivacity = \frac{1}{P} \frac{dP}{dt}$$

We calculated the vivacity measured from deflagration of pristine PBXN-109 and from deflagration of the thermally-damaged samples. For data taken around the same pressure with burning pressure exponents close to one, the vivacity is proportional to the surface area involved in the deflagration. Therefore, the ratio of vivacities of damaged to pristine samples give an estimate of the increase in surface area in the damaged samples. We have made preliminary surface area calculations for these experiments and the results are shown in Figure 6. We are working with Dr. Patrick Baker and his colleagues at the Army Research Laboratory to improve our vivacity calculations, but in the meantime the results are illustrative of the effect of thermal damage. We see that the initial surface area is higher by up to a factor of 20 in comparison with pristine material. This is consistent with the 10-20 fold increase in deflagration rate shown in Figure 4, and indicates that the increase in burning rate is essentially caused by the increase in surface area, not by acceleration of chemical reactions at the higher initial temperatures. The kinks in the curves in Figure 6 are an artifact of our non-optimized method to calculate vivacity, but ignoring them we clearly see that the increase in surface area drops off rapidly as pressure increases. As stated above, we do not understand this behavior, but it is consistently seen in all runs.

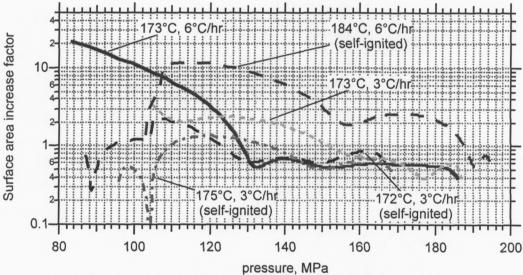


Figure 6. Increase in surface area for thermally damaged PBXN-109, as calculated from vivacity of pristine and thermally damaged samples.

COMPOSITION B

Composition B exhibited deflagration behavior unlike that of other materials that we have tested. In virtually every case, the first few pellets burned relatively slowly and with apparent uniformity, but later pellets burned very rapidly and erratically. This behavior is seen in the pressure-time data in Figure 7 and in the burn rates calculated from the burn wire data, shown in Figure 8. There is a large scatter in the data taken during the very high burn rates seen in the later pellets in each run. Nonetheless, the first few pellets do provide a measurement of the burn rate of Composition B before it undergoes the transition to rapid deflagration. A fit to these first few points for each run is also shown in Figure 8. As also shown in

Figure 8, there was no apparent effect of either the sample preparation (cast or pressed) or the inhibitor on the cylindrical surface of the pellets (polyurethane or epoxy-coated polyurethane).

We see two interesting features in Figure 8. First, the fit to the lower edge of the data set, representing the burning of the first few pellets, shows a second-order pressure dependence, very high compared with previous results with HMX-based explosives⁸ and AP-based propellants¹ and current results with PBXN-109. Second, the onset of rapid burning, as also seen in Figure 7, presumably represents a deconsolidation of the sample during the deflagration process. Estimation of the increase in surface area during deconsolidation using the measured vivacity is currently underway and will be reported in future work.

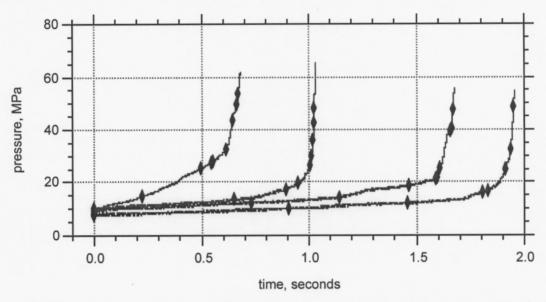


Figure 7. Typical pressure data for Composition B in the strand burner. The blue symbols show the flame arrival time as indicated by the embedded burn wires.

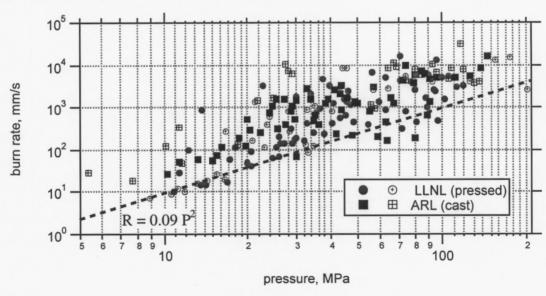


Figure 8. Burn rate data for Composition B, using pressed and cast samples. The dotted line shows a fit to the lower edge of the data set, and the parameters for the fit are also listed. Hollow symbols represent runs with polyurethane inhibitor, and solid symbols represent runs with epoxy coating over polyurethane.

The high pressure dependence of Composition B deflagration has been previously observed. Birk and coworkers at ARL reported results from interrupted burning tests at pressures up to 70 MPa, and found an overall pressure dependence of 1.712.⁵ Later work by the same group showed the pressure exponent ranging from 1.5 to 1.7 in measurements with a strand burner at 300-1,500 psi (2-10 MPa) and with a closed bomb at 1,500 – 15,000 psi (10-100 MPa) ⁴ We will continue our efforts to understand our data in the context of these results under different conditions.

With Composition B, the first few pellets burn with apparent uniformity and the following pellets burn rapidly, regardless of the initial pressure. Therefore the deconsolidation process in Composition B seems to be time dependent instead of pressure dependent as in HMX-based explosives. This may be a result of TNT in the high-temperature environment of the pressure vessel — the first few pellets may burn uniformly before the heat can penetrate and melt the TNT, leading to subsequent mechanical failure and exposure of the RDX crystal surface to flame. We have experiments underway to check this idea — by heating samples to around 100°C, we will melt the TNT before ignition. If we do not see deconsolidative burning with these samples, we can conclude that the melting process does contribute to the deconsolidation.

RAMIFICATIONS WITH RESPECT TO THERMAL EXPLOSION VIOLENCE

It has been observed that Composition B gives quite violent thermal explosions, ⁹ whereas PBXN-109 yields much less energetic reactions. ^{7, 10, 11} The pressure dependence of the deflagration rates at high pressures at least partly explains their relative responses. For materials with a low pressure dependence of high-pressure deflagration, such as PBXN-109 (n=0.85), the reaction rate is accelerated at a relatively low rate. This leads to relatively low violence of thermal explosion, as has been observed in the NAWC thermal explosion experiments with PBXN-109. In contrast, for a material with a high pressure dependence for deflagration, such as Composition B (n=2), the high-pressure deflagration is accelerated at a very high rate, leading to very violent thermal explosions such as those seen in LLNL STEX results. In addition, the deconsolidation leading to high deflagration rates seen with Composition B should drive the violence even higher. Therefore, the measurement of high-pressure deflagration behavior for any material provides immediate qualitative insight as to the expected violence of thermal explosion, even before the application of these results in predictive models.

SUMMARY AND CONCLUSIONS

The deflagration behavior of pristine PBXN-109 is a smooth function of pressure, with the pressure exponent decreasing from 1.32 to 0.85 at pressures above 20,000 psi (135 MPa). When thermally degraded under conditions replicating those in actual cookoff experiments, the deflagration rate increases by a factor of 10-20, apparently as a result of increased surface area.

In contrast, Composition B exhibits rapid and erratic deflagration following an initial period of well-behaved combustion, regardless of initial pressure. Even the initial deflagration is unusual in that the rate is second-order in pressure. The onset of the rapid and erratic deflagration may be driven by heat flow into the sample causing the TNT to melt, with subsequent deconsolidative failure of the sample and greatly increased surface area. Experiments are underway to determine if this is the case.

In both cases, it appears that rapid deflagration behavior is driven by increases in surface area available during the burning process. Calculation of vivacity allows us to estimate the increase in surface area, and this has been done for PBXN-109 data. This calculated increase in surface area can be directly applied in deflagration models in computer simulations of thermal explosions, thereby providing a means to incorporate the effect of thermal damage on deflagration rates into these simulations.¹²

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